

DETECTION OF INTERGRANULAR CORROSION IN COLD PLATE FACE SHEETS

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Abstract

Cold plates are critical for cooling electronic systems in the shuttle. As a result of the environmental conditions in which they operate, water can condense between them and a support shelf. In some cases, this water results in intergranular corrosion in the face sheet. If the intergranular corrosion sufficiently penetrates the face sheet, a coolant leak could occur and jeopardize cold plate operation. This paper examines techniques for detecting and characterizing the intergranular corrosion, to enable re-certification of cold plates that have been in operation for 15 plus years.

Intergranular corrosion was artificially induced in the face sheets of a series of cold plate specimens using an electrochemical process. Some of the cold plate specimens were separated for destructive characterization of the extent of corrosion produced by the electrochemical process and to insure the induced corrosion was intergranular. The rest of the specimens were characterized nondestructively using several techniques. X-ray tomography and ultrasonic techniques provided the best indication of corrosion in these specimens and will be the focus of this paper.

An x-ray tomography technique was shown to be the most effective technique for characterizing depth of the intergranular corrosion. From these measurements, corrosion profile maps were developed that were consistent with subsequent destructive evaluations of the specimens. This enabled the assessment of NDE standards to evaluate the viability of other NDE techniques. Due to system constraints, a different technique must be used to inspect an entire cold plate.

An ultrasonic technique was shown to be very reliable for detection of corrosion in the unbacked regions of the face sheet. The ultrasonic technique was performed in an alcohol bath to avoid additional corrosion during the NDE evaluation. A pulse echo technique that focuses on the RMS value of the signal is shown to be very sensitive to the presence of intergranular corrosion.

Introduction

During a maintenance cycle of the Shuttle Orbiter (OV-102), helium leak detection of an avionics cold plate identified a leak located in the face sheet oriented towards the support shelf. Subsequent microscopic examinations of cross sections of the face sheet of the leaking cold plate revealed that intergranular corrosion had penetrated the 0.017-inch thick aluminum (AA6061) face sheet. The intergranular attack (IGA) is likely caused by an aggressive crevice environment created from condensed water vapor located between the cold plate and support shelf. To help to assess the current state of other in-service cold plate, it was necessary to develop standards representing different extents of corrosion and a nondestructive technique capable of detecting the intergranular corrosion.

Significant efforts have been made to detect and characterize crevice corrosion in aircraft structures. Much of this work has focused on specimens fabricated to represent back surface material loss in the crevice between two plates. A main driver for this work is the relative ease that samples can be prepared for testing the sensitivity of different techniques. Before assembling a two or three layer

specimen, the hidden surfaces of the plates can be machined to reduce the thickness of the plates by known amounts. This provides a well-characterized set of samples with known extents of material loss.

By comparison, relatively little effort has been made to develop techniques for detecting and characterizing the extent of intergranular corrosion, where the grains remain in place and there is no significant material loss. Compared to material loss specimens, accurate specimens that represent this type of corrosion are relatively difficult to fabricate. After the specimens have been fabricated, it is difficult to characterize the extent of corrosion without destroying the specimens.

This paper presents an accelerated corrosion method for fabricating specimens with corrosion representative of the corrosion found in the orbiter's cold plates. To assess the extent of the corrosion in the cold plates, microfocus x-ray tomography was performed on the corroded specimens. The microfocus tomography system is only capable of imaging small specimens, not a full cold plate. To detect the presence of corrosion with a technique capable of inspecting entire plates, a pulse echo ultrasonic technique was developed. A comparison of the results obtained with ultrasonics and the computed tomography are presented.

Description of the Cold Plate

A diagram showing a typical region of the cold plates is shown in Figure 1. The largest area of the cold plates consists of three separate sheets separated by posts. The cooling fluid flows around the post and is contained between the two face sheets. The cold plates are fabricated from aluminum (AA6061). During fabrication, a 1000°F brazing process was used to bond the face sheet to the posts of the cold plates. Following the brazing, the cold plate components are slowly cooled to avoid distortion of the bonded cold plate. This slow cooling process causes excessive grain boundary precipitation resulting in a material that is susceptible to intergranular attack (IGA). The IGA is likely caused by an aggressive crevice environment created from condensed water vapor located between the cold plate and support shelf.

The specimens used in this effort were obtained by sectioning a portion of one of the flight hardware cold plates that had been taken out of service¹. This portion was sectioned into specimens approximately 1 inch by 1 inch. This size optimized the use of the cold plate and produced specimens that could easily be imaged with the microfocus tomography system. Some of the specimens used in this effort are shown in the photograph in Figure 1.

¹ The cold plate was removed from Orbiter OV-102. The cold plate passed the helium leak detection procedure and visual inspection detected no evidence of corrosion.

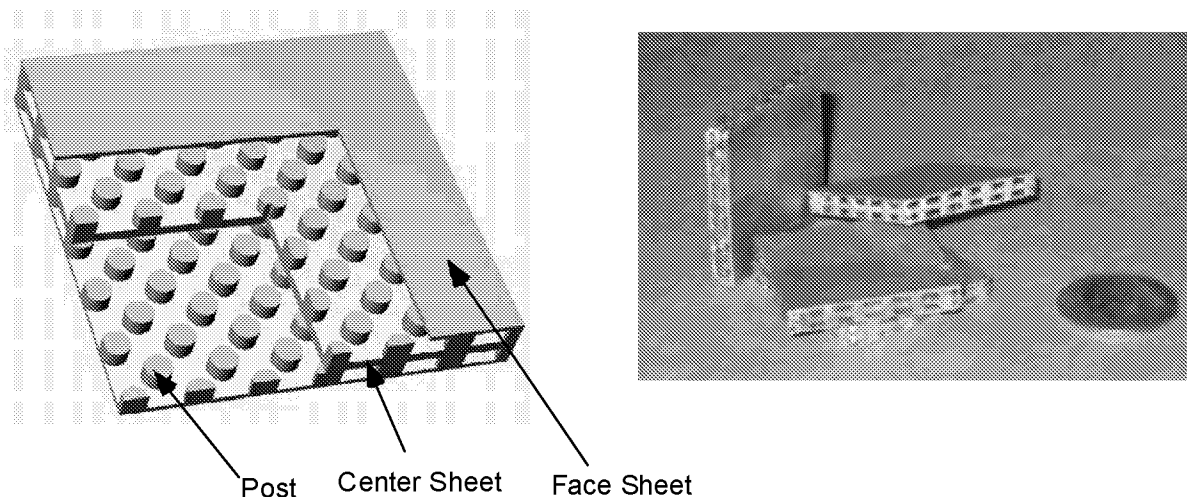


Figure 1. Diagram of typical portion of the specimen and photograph of several of the specimens with induced corrosion.

Accelerated Corrosion Method

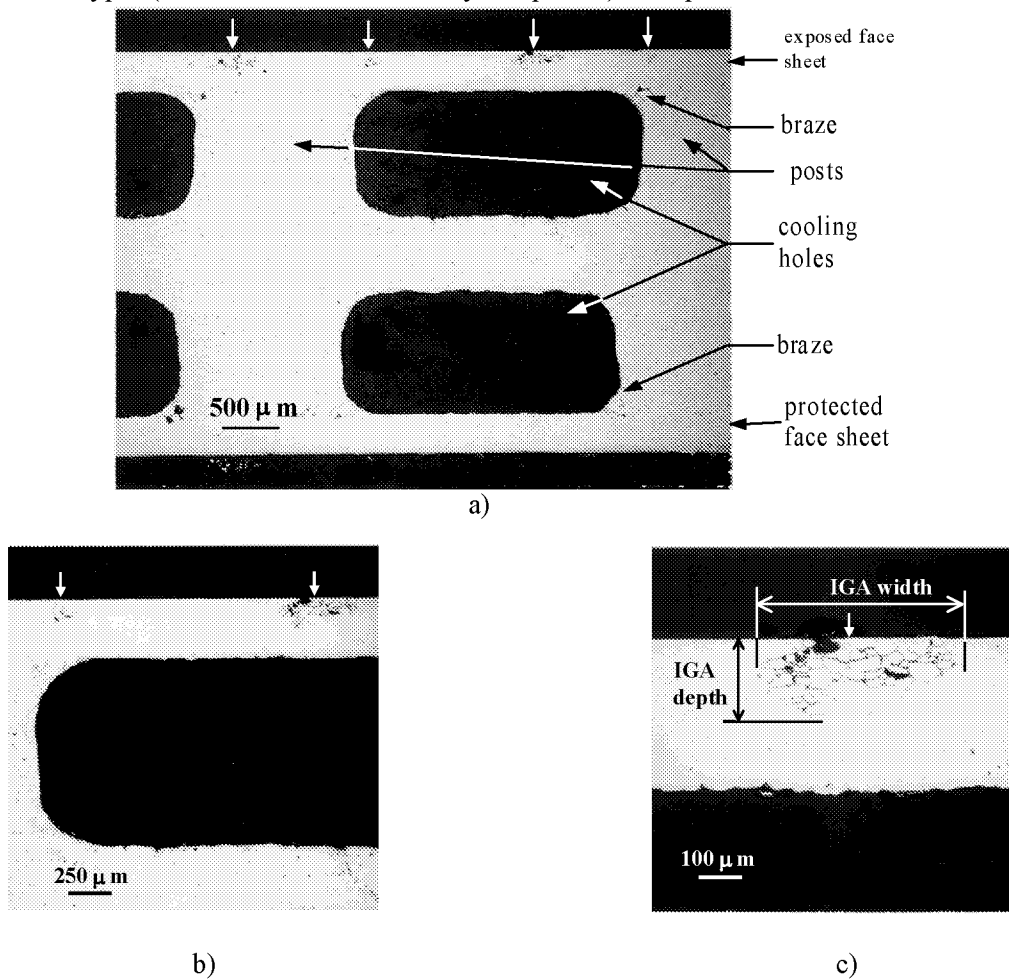
Test specimens and NDE standards (1-inch by 1-inch) were machined from a cold plate and prepared in the following manner. One face of the specimen was polished (thru 600 grit) to remove the protective chromate film applied during manufacture and to expose the bare face sheet material. All specimen surfaces, except for the polished face sheet region, were masked with a corrosion protective wax. Specimens were electrically coupled to a cathode (steel coupon) and immersed in a mild electrolyte solution (20 mM - NaCl, 4mM - NaNO₂, 4 mM - NaHCO₃, 2 mM - NaF). The cathode was used to supply a corrosion current to the 1 square inch anode (polished cold plate surface) and thus accelerate the intergranular corrosion. A series of preliminary electrochemical experiments were performed; here, the surface area of the cathode and the exposure time were varied until the optimum amount of IGA was produced in the exposed face sheet. Two different cathodes were used, a 1-1/2-inch by 1-1/2-inch and a 2-1/4-inch by 2-1/4-inch steel electrode. By changing the exposed surface area of the cathode, the applied corrosion current was varied. When coupled to the 1-1/2-inch by 1-1/2-inch cathode, the electrochemical potential of the cold plate specimens was \approx -600 mV versus a saturated calomel electrode (SCE). Exposure time was varied between 8 and 30 hours.

Destructive Examination of Specimens with Induced Corrosion

Destructive examination results were used to qualify the laboratory IGA corrosion test method. To accurately characterize the IGA produced by the laboratory test method, specimens were metallographically sectioned through the thickness, mounted edge-on in epoxy, polished and examined using an optical microscope. Selected regions containing IGA were examined in great detail; here, multiple metallographic sections at 0.004 to 0.008-inch intervals were performed to accurately characterize the corrosion events. Each metallographic section was examined at high magnification to determine the maximum IGA depth, width and position.

Initial destructive examinations were performed on corroded cold plate samples to evaluate the morphology of the IGA and to compare these results with the IGA observed in cold plates removed from the orbiter. Figure 2 contains a series of optical micrographs for a corroded sample that was exposed to the electrolyte solution for 24 hours, sectioned and polished. Several discreet corrosion events, identified by arrows in Figures 2a and b, are identified along each section. Here, the 1-inch by 1-inch cold plate

sample was coupled to a 1-1/2-inch by 1-1/2-inch steel cathode and immersed in the electrolyte. The resultant IGA was very similar to that observed for in-service cold plates. For each event, a maximum penetration depth and width were recorded (see Figure 2c). Such measurements were performed for several specimens exposed to the electrolyte solution for various times. Figure 3 shows results of depth versus width of IGA for three specimens exposed to the electrolyte for 8 hours (Figure 2a) and three specimens exposed for 24 hours (Figure 2b). Each specimen was polished six times, to examine six different cross-sections. Microscopic measurements of width, depth and position for all IGA events were performed on each metallographic section, to provide an accurate assessment of the IGA events. As can be seen in Figure 3, the average depth and width for the observed IGA was greater when the cold plate samples were exposed to the solution for 24 hours (see Figure 3b) than when they were exposed for 8 hours (see Figure 3a). To ensure that the NDE standards contained a wide distribution of IGA events, two NDE standard types (24 hour and 8 hour electrolyte exposure) were produced.



Figures 2 a-c. Optical micrographs for three different magnification of IGA produced in a cold plate specimen in the laboratory. The 1-inch by 1-inch specimen was coupled to a 1-1/2-inch by 1-1/2-inch steel cathode and exposed to the electrolyte solution for 24 hours.

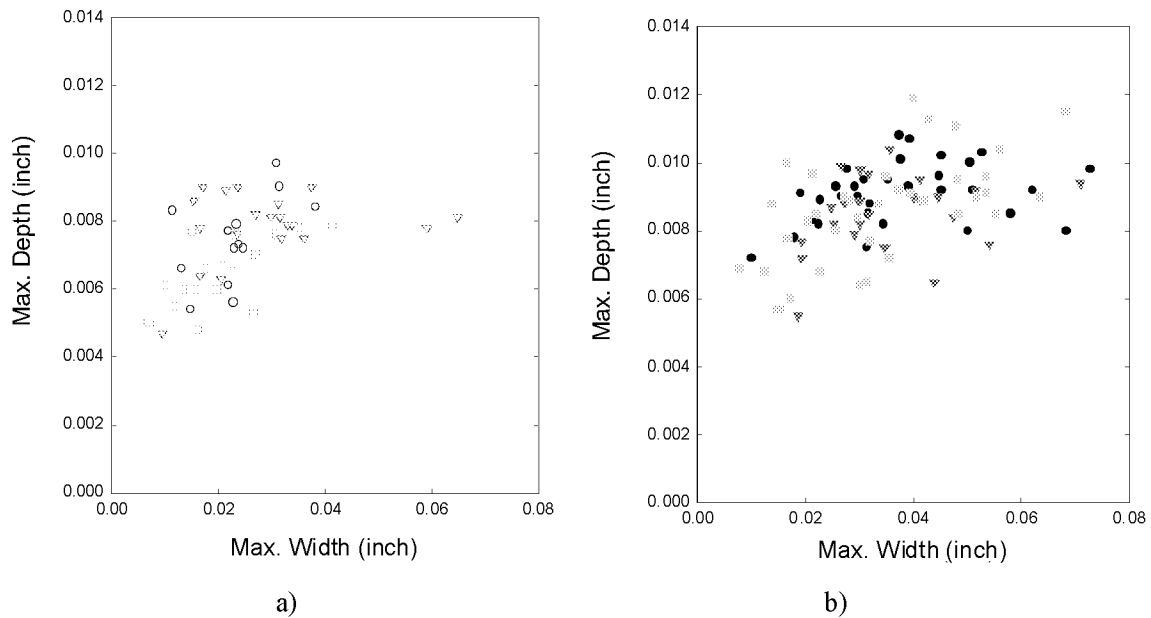


Figure 3. Plots showing depth versus width of IGA events measured using optical microscopy for cold plates specimens exposed to the electrolyte for a) 8 hours and b) 24 hours.

CT Radiography (Microfocus X-ray Computed Tomography)

Computed tomography was performed with a microfocus CT system. The microfocus x-ray source is 160 kV with a 0.002-inch spot size. The detector package contains 8192 separate detectors with 0.001-inch center separation and was positioned approximately 40-inches from the source. The specimen was mounted on a rotation stage and was centered between the source and detector. Geometric constraints limit the field of view of the system in the horizontal plane to a circle approximately 3.5-inches in diameter. The x-ray attenuation of aluminum was the limiting factor for measurements in these specimens. Specimens were limited to 1-inch by 1-inch to obtain a reasonable signal to noise ratio when using the 160 kV source.

Data was acquired every 0.1 degrees of rotation for each specimen to obtain the required information for computed reconstruction of the density for a specimen cross-section. At each rotation angle, the data was acquired by integrating the response of the detectors for 10 seconds. Including data transfer time, the total time required to acquire a single cross section was approximately 11 hours.

The microfocus x-ray system was capable of generating images with voxels 0.0005-inch by 0.0005-inch with a vertical height of 0.004-inch. For such images, resolution in the horizontal plane was 0.0025-inch by 0.0025-inch. The rotation stage was mounted on a vertical positioning stage; the position of the vertical stage specifies the portion of the depth into the specimen that will be imaged. A series of images with different mean sampling depths were acquired by moving the vertical stage in steps of 0.002-inch. This results in overlaps of 50% between data sets. A typical set of images obtained for different depths is shown in Figure 4. The IGA events are visible as regions of slightly darker intensity. As the samples are obtained from distances further from the exposed surface, the number of events decreases until no events are detectable.

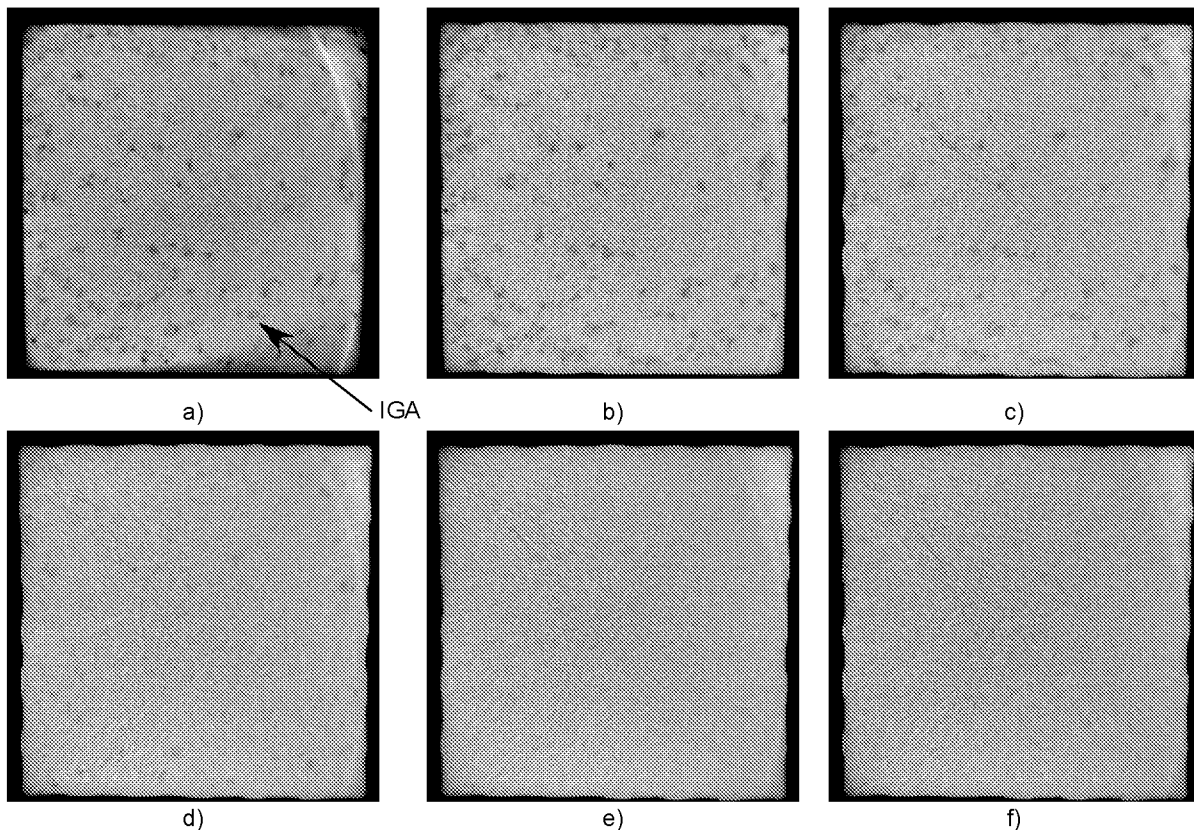


Figure 4. CT radiography images for a cold plate sample exposed to an electrolyte solution for 24 hours. The sensor head was located at a height that results in a mean sampling depth of 0.0135-inch. Average sampling depths are a) 0.0035-inch, b) 0.0055-inch, c) 0.0075-inch, d) 0.0095-inch, e) 0.0115-inch and f) 0.0135-inch

To validate the CT radiography results, metallography was performed to confirm IGA corrosion depth measurements. For each image in Figure 4, the suspected IGA events were identified in the images. The events identified in each image are shown in Figure 5. Because discrete depth images were produced, a mean maximum depth was determined for each IGA corrosion event. For example, a corrosion event is identified in region A in Figure 5a. Here, the event was observed for the 0.0035 and 0.0055-inch images (Figure 5 a and b) and was not observed at a depth of 0.0075-inch and greater (Figures 5 c-f). For this example, a mean maximum IGA depth of 0.0065-inch was determined for this event. Similarly, an IGA event would be determined to have a mean maximum depth of 0.0045-inch if it was identified at a depth of 0.0035-inch and not observed at the 0.0055-inch depth. If an event were identified in only one CT image, the event would be discarded from further consideration.

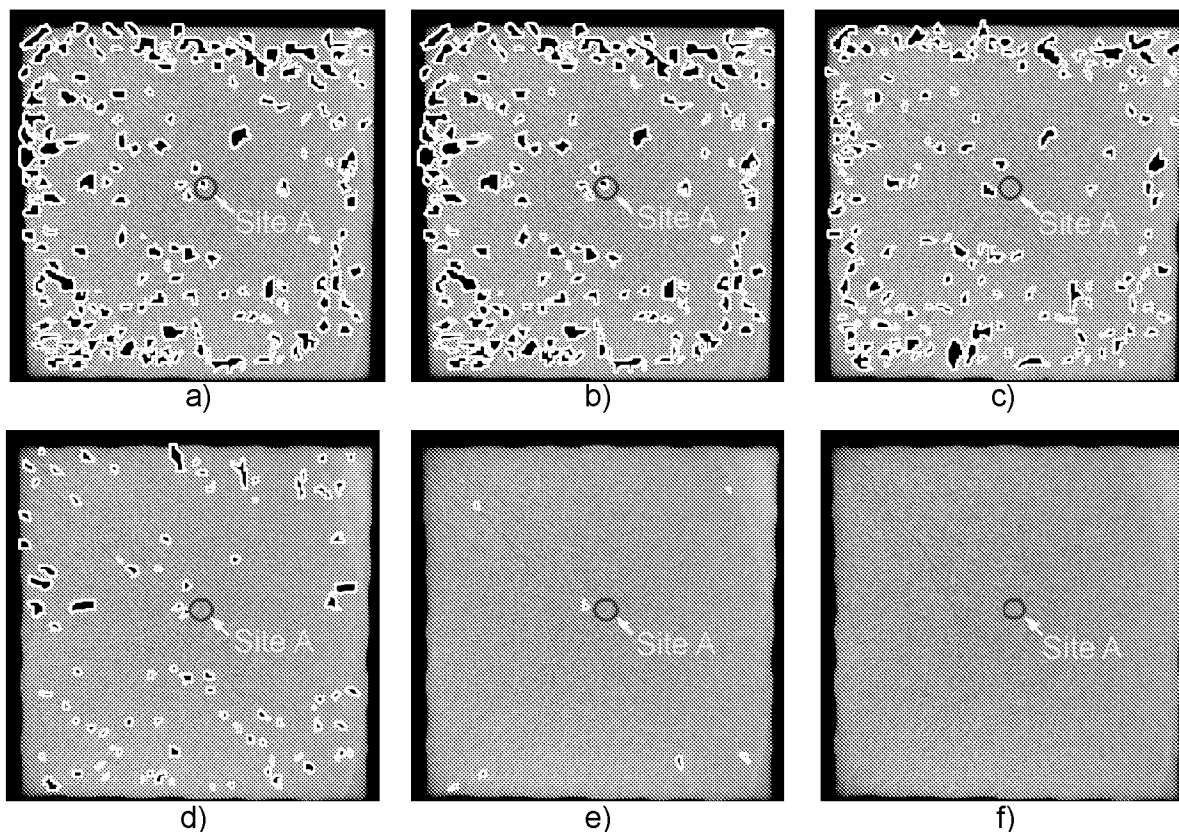


Figure 5. CT radiography images for a cold plate sample exposed to an electrolyte solution for 24 hours with IGA events identified. Average sampling depths are a) 0.0035-inch, b) 0.0055-inch, c) 0.0075-inch, d) 0.0095-inch, e) 0.0115-inch and f) 0.0135-inch. f) has no identified events.

Two additional corroded (24 hour) specimens were examined to determine the accuracy of the CT radiography system in measuring IGA. The CT radiographic inspections were conducted at the same depths describe above; thus categorizing the IGA events at mean maximum corrosion depths of 0.0045, 0.0065, 0.0085, 0.0105 or 0.0125-inch. After CT radiography of the two corroded specimens was completed, a total of 32 metallographic cross-sections were microscopically examined. Each IGA corrosion event was characterized for depth, width and location and compared to the CT radiography results. Figure 6 is a plot of the mean maximum depth for 27 IGA events determined by CT radiography versus the maximum depth of the same events determined by the destructive examinations. Table I summarizes the statistical analysis of the data presented in Figure 6. A 95% confidence interval was determined for CT radiography results at the 0.0065, 0.0085, and 0.0105-inch mean maximum depths. If CT radiography indicates that an IGA event is 0.0105-inch deep, there is a 95% certainty that the event is between 0.0086 and 0.0122-inch in depth. Due to a lack of data, it was not possible to determine confidence intervals for CT results less than 0.0065-inch and greater than 0.0105-inch. Based on these results, the CT radiographic method enables nondestructive characterization of the IGA corrosion samples and provides good standards for development of an inspection technique.

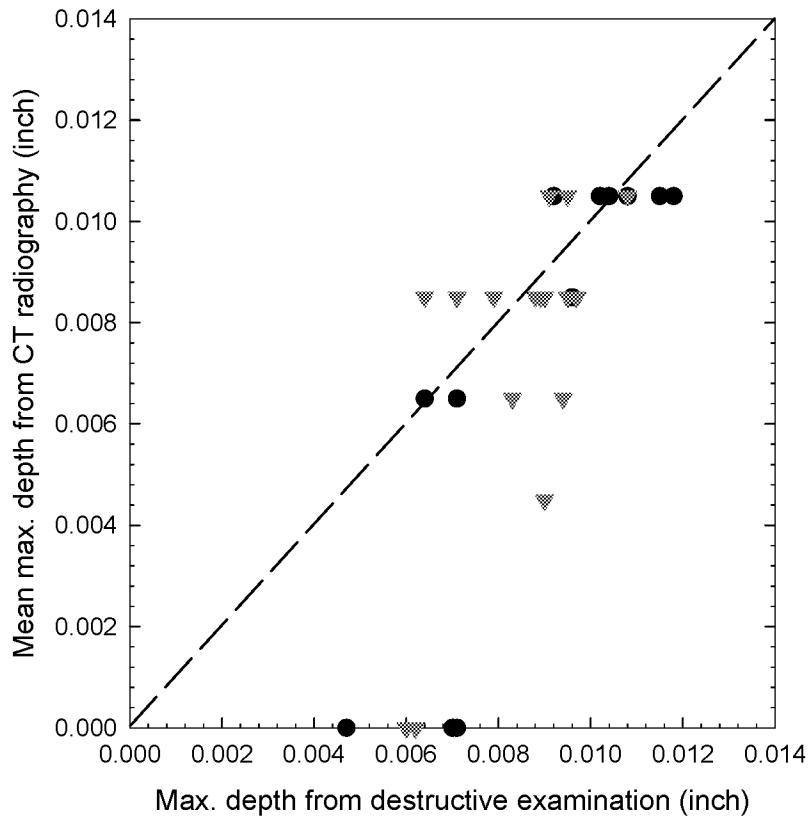


Figure 6. Plot of mean maximum depth of IGA events as determined by CT radiography versus maximum depth of the same IGA events as determined by a destructive examination for two cold plate samples exposed to an electrolyte solution for 24 hours while coupled to a steel cathode.

Table I. Confidence intervals for each of the maximum IGA depths identified by CT radiography for cold plate specimens exposed for 24 hours.

Mean max. depth from CT radiography (inch)	95% confidence interval (inch)
0.0045	--- *
0.0065	0.0058 – 0.0105
0.0085	0.0068 – 0.0106
0.0105	0.0086 – 0.0122
0.0125	--- *

* - Confidence intervals for 0.0045 and 0.0125-inch CT results could not be determined due to insufficient data correlating CT results with destructive evaluation.

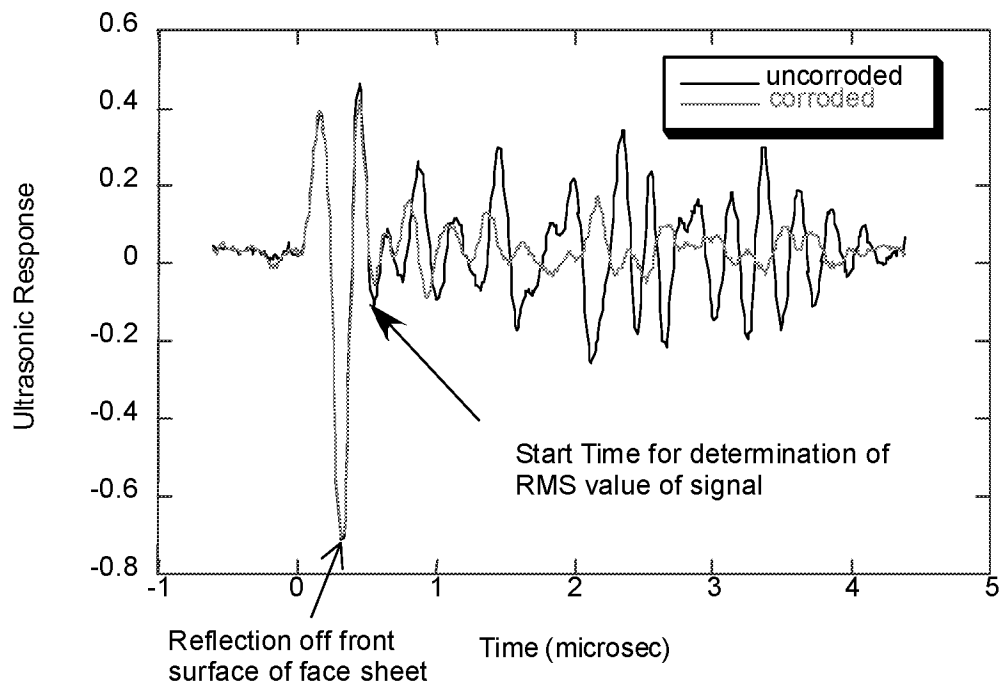


Figure 7. Ultrasonic response for immersion ultrasonic analysis for uncorroded and corroded cold plate samples.

Pulse Echo Ultrasonics Technique for Detection of IGA Events

The maximum size of a specimen that can be inspected with the x-ray microfocus tomography system is much smaller than the size of the cold plates. Therefore, inspection of an entire cold plate required development of an alternate inspection system. Several different techniques were applied to the standards. The most successful technique for detection of individual IGA events was pulse echo ultrasonics.

A specimen was placed in a scan tank with alcohol used as the couplant fluid between the transducer and specimen. Alcohol was used as the couplant to insure no addition corrosion occurred during the inspection process. A 20 MHz focused transducer was scanned above the specimen surface and the ultrasonic response was recorded digitally. To ensure that the face-sheet portion of the specimen was placed at the proper transducer focal length, a thin wire was placed on the surface of each specimen before performing any measurements. A sharp image of the wire was acquired by adjusting the vertical position of the transducer above the specimen. The transducer was scanned over the area in 0.01-inch increments. At each position, the transducer was excited with a short, broadband electronic pulse, which produced a broadband ultrasonic wave that propagated to the specimen. The echo of the sound wave was measured with the same transducer. This measurement was digitized at 100 MHz and recorded for post processing. The digitization was triggered by a signal corresponding to the echo off the front surface of the specimen. The digitizer was configured to enable capture and digitization of the output of the transducer both before and after the trigger is received.

Typical responses for corroded and uncorroded points on the specimen are shown in Figure 7. The initial response at both points is a strong reflection off the front surface. A frequency analysis indicates the measurement system bandwidth was approximately 3 to 6 MHz. This is too narrow to enable discrimination of the individual echoes from the back surface of the face sheet, which has a resonance

frequency of approximately 6 MHz. However, the RMS value of the ultrasonic response following the front surface echo is clearly less for the corroded point, than for the uncorroded point.

To image the corrosion, the RMS value of the ultrasonic response between 1 and 4 microseconds was calculated from the digitized waveforms. From these values it is possible to create an image such as shown in Figure 8. For comparison, a microfocus tomography image of the same specimen is also shown. Where there are posts below the face sheet, there are no echoes within the gated time, and the RMS value of the signal is small. This yields the round dark regions that are clearly visible in the image. Unlike the CT radiography technique, immersion ultrasonics does not provide a direct method for evaluating the penetration depth of the IGA events. An exact comparison of the ultrasonic and CT radiography was not possible due to difficulties registering the two images. However, qualitative agreement between the two images is reasonable. Future efforts will focus on a more quantitative comparison.

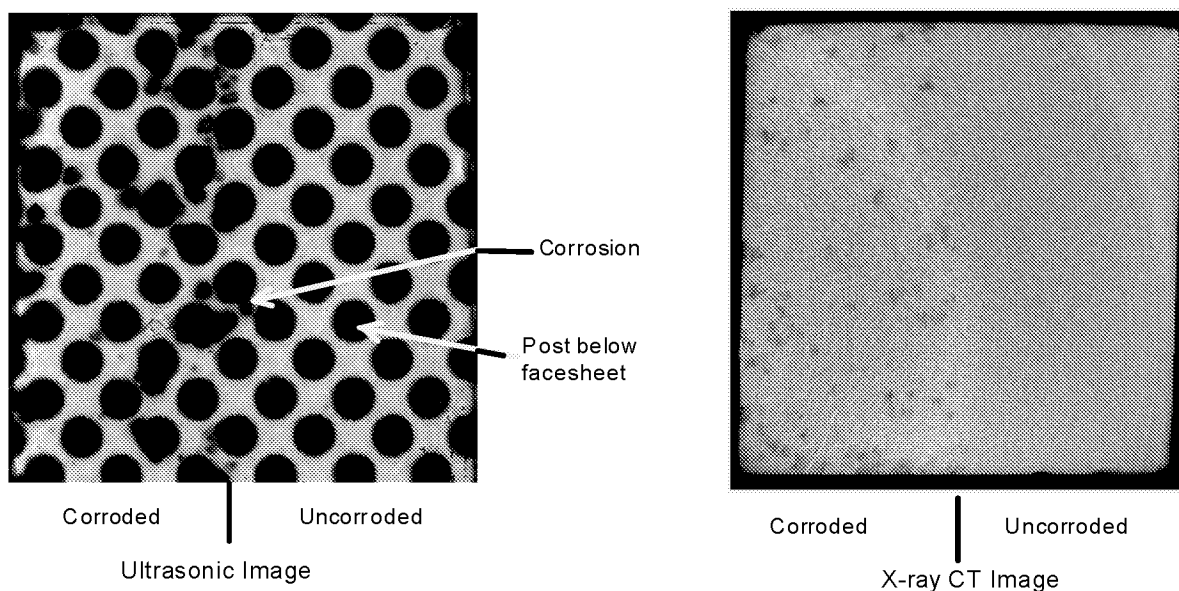


Figure 8. Immersion ultrasonic results for a corroded cold plate sample. The right half of the specimen was coated with wax to prevent corrosion, while the left half of the specimen was exposed to an electrolyte solution for 24 hours while coupled to a steel cathode.

Summary

An accelerated laboratory method was developed that reproduces in-service IGA corrosion observed in avionic cold plates. This accelerated technique was used to produce NDE standards that contain IGA. CT radiography was successfully used to fully characterize depth of the intergranular NDE standards after detailed metallography was conducted to validate CT radiography results. Immersion pulse echo ultrasonics is shown to be capable of detecting the intergranular corrosion. It has the resolution to delineate discrete corrosion events while being able to inspect large components.